UNIVERSITY OF PENNSYLVANIA

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A THESIS

IN PHYSICS

PRESENTED TO THE FACULTY OF THE GRADUATE SCHOOL IN
PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR
THE DEGREE OF DOCTOR OF PHILOSOPHY

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FINE STRUCTURE OF THE *K*-RADIATION OF THE LIGHTER ELEMENTS

BY LAWRENCE Y. FAUST
RANDAL MORGAN LABORATORY OF PHYSICS, UNIVERSITY OF PENNSYLVANIA

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ABSTRACT

A vacuum spectrograph of simple construction is described in which it is possible to maintain bombarding currents of 150 milliamperes. With a line grating on glass (1179 lines per mm) photographs, some measurable in the second and third orders have been obtained of the soft x-radiation from beryllium, boron, oxygen and carbon, also from tungsten and certain heavier elements. Under analysis with a thermo-electric densitometer the *K* lines of the lighter elements, excepting oxygen, were found to be complex. The *K*-radiation of boron compounds contained 13 components, that of "pure" boron four, that of carbon nine whereas the beryllium radiation was still more complex. The relative intensities of these components was shown to depend on the energy used in stimulating them. The representative wave-length selected for the $K\alpha$ line was for oxygen 23.7A, for boron 69.3A, for carbon 45.3 while for beryllium the radiation embraced more than 15A with maxima at 107.2, 113.2 and 118.7A. The complex structure is thought to be partly a true fine structure and partly a system of satellites due to interaction with associated dissimilar atoms. A possible hypothesis is suggested in explanation of these results.

INTRODUCTION

THE possibility of diffracting soft x-rays from line gratings at small glancing angles was first pointed out by Professor A. H. Compton,[1] in 1923. Since that time important advances have been made, especially by Thibaud,[2] in the technique of this method, resulting in a number of valuable contributions to our knowledge of the frequencies and inter-relations of spectral lines in the soft x-ray region. Thibaud[3] has measured the *K* lines of carbon, oxygen, nitrogen and boron and the *L*, *M*, and *N* radiations of several elements of high atomic number. Other investigators including Hunt,[4] Osgood,[5] Weatherby[6] and Howe[7] have concentrated on the measurement of the *K* line of carbon in view of its possible value as a calibrating or reference line. Söderman[8] has made careful measurements of the *K* lines of the elements between magnesium and beryllium with the exception of neon. In addition to these wave-length measurements Bazzoni[9,10] Faust and

[1] A. H. Compton, Phil. Mag. **45**, 1121 (1923).
[2] Thibaud, Journ. de Phys. et le Rad. **1**, 13 (1927).
[3] Thibaud, Comptes Rendus, Jan., 1926. Thibaud, Phys. Zeits. **28**, 241 (1928).
[4] Hunt, Phys. Rev. **30**, 227 (1927).
[5] Osgood, Phys. Rev. **30**, 567 (1927).
[6] Weatherby, Phys. Rev. **32**, 707 (1928).
[7] Howe, Proc. Nat. Acad. Sci. Mar., 1928.
[8] Söderman, Zeits. f. Physik **52**, 795 (1929).
[9] Bazzoni, Faust and Weatherby, Nature, May 11 (1929) p. 717.
[10] Bazzoni, Faust and Weatherby, Abstract, Am. Phys. Soc., Washington Meeting, Apr., 1929, Phys. Rev. **33**, 1101 (1929).

Weatherby have studied the fine structure of the K lines of carbon and boron by densitometric methods.

It is evident that the line grating offers amongst other advantages the best means for investigating the K-radiation and associated K-absorption edges of the light elements. There is some reason to suspect that with the light elements the proximity of the K level to the exterior of the atom may cause a deviation from the simplicity of structure characteristic of K lines of the heavier elements. However, the researches listed above were for the most part carried out with too low resolving powers to bring out any fine structure in the K lines if such structure existed. The single exception to this statement is in the work of Bazzoni, Faust and Weatherby who densitometered the lines obtained in the first, second and third orders from a grating of 1179 lines per mm. These workers reported that the K line of carbon contains four components lying between 44.2, 44.9, 45.4 and 46.2A and that the K line of boron is of still more complex construction.

The investigation reported on in the present paper was undertaken to check in greater detail the measurements reported by these last mentioned authors and to extend such measurements to other elements of low atomic number.

The Apparatus

The apparatus consisted of a special vacuum spectrograph constructed of brass pipe in a design similar to that of Weatherby.[11] This apparatus differed from Weatherby's in several particulars. In the first place it was of larger diameter permitting the use of a photographic plate 5″ long at a distance of 40 cm from the grating. In the second place the anode-cathode structure was altered to make closer adjustment of clearances possible and to obtain a higher electron current. Both anticathode and filament were mounted on standards attached to a single plate which was wax-sealed to the spectrograph. A spiral filament was centered in an adjustable steel focusing ring coaxial with the anticathode. This ring, which formed one of the filament leads, was earthed and was separately water-cooled by a jacket surrounding the anticathode and insulated therefrom by a Pyrex tube. The other filament lead and the anticathode itself were also each water-cooled and were suitably isolated electrically by Pyrex insulation. The maximum clearance between the cathode and anticathode structure did not exceed 1/8 inch. This design incorporates the advantages of Dershem's[12] tube but is much simpler.

In order to prevent the fogging of the photographic plate as much as possible, carbon paper was introduced into the path of the direct and reflected beams. Considerable scattering resulted from the use of carbon paper, and later aluminum foil was substituted. This substitution resulted in producing clearcut, sharply measurable images of the direct and reflected beams.

[11] Weatherby, reference 6.

[12] Dershem, Journ. Optical Society of America **18**, 127 (1929).

Eastman x-ray plates were used in all the work with exposures varying from 15 minutes to six hours, three hours being the average.

The current to the anticathode was usually approximately 60 m.a. The driving potential was obtained from d.c. generators in series. A vacuum as low as 10^{-4} mm could be obtained in the spectrograph during operation by the action of a set of diffusion pumps.

The width of the two slits used in collimating the x-ray beam was 0.1 mm or less. The width of the grating used was 2 mm.

At this point attention may be called to the fact that Thibaud[2] has investigated the collimating properties of a grating at grazing angles. He obtains a formula for the variation of diffraction angle with the grazing angle.

$$\frac{di}{d\theta} = \frac{\theta/2}{(2n\lambda/d + (\theta/2)^2)^{1/2}}.$$

Thus when $\theta = 0$ the diffracted beam is parallel. The maximum variation of θ in this work due to the width of the grating and the width of the slits was $\Delta\theta = 5'$. Whence for a grating of 1179 lines per mm with an angle 3° at 50A, $di/d\theta = 0.2$. Thus corresponding to 5′ for $\Delta\theta$ we have 1′ change in i which corresponds at 50A to about 0.08A or about the experimental limit of accuracy. In general the values of θ and $\Delta\theta$ were about half the above values whence the error would be roughly 0.02A which is well within experimental error. It is therefore apparent that in the apparatus as used in this work errors due to lack of perfect collimation of the beams were negligible.

Measurements

The determination of the wave-length of a line is dependent (Fig. 1) on the measurement of the angles α and θ and the grating constant d used in the formula:

$$n\lambda = 2d \sin(\alpha/2) \sin(\alpha + \theta)/2.$$

The constant d, obtained by the usual procedure employing the 5461 line of the Hg arc was found to be 8481.8A corresponding to a ruling of 1179 lines per mm. The grating used was furnished by Professor R. W. Wood, to whom we hereby acknowledge indebtedness. Tan θ can be obtained by

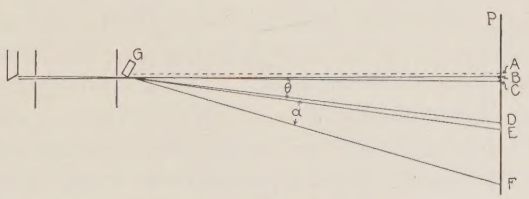

Fig. 1. Diagram of apparatus.

dividing the distance between the direct beam (BC. Fig. 1.) of x-rays passing by the grating and the beam (DE) reflected from the surface of the grating by the perpendicular distance (GA) from the effective center of the grating to the photographic plate.

The measurement of the distance GA was made on a comparator by comparison with a standard meter. The accuracy of this measurement was within 0.05 mm in 40 cm. Since care was taken to insure that the entire face of the grating was in the path of the incident beam, this measurement should correspond to the equivalent distance between grating and plate.

The distance BC to DE was found from a direct measurement on a Hilger traveling micrometer microscope of the extreme edges of the direct and reflected beams corresponding to the distance BE. This distance divided by the distance, GA, of the plate from the grating was taken as the corrected value for tan θ. An examination of Fig. 1 shows the justification of this procedure. BC is the direct beam image as found on the plate. In the absence of the grating this beam would extend from C to A. Thus AB is the position of the direct beam cut off by the grating and is therefore the effective direct beam. Then $BD+AB/2+DE/2$ represents the real distance between the reflected beam and effective direct beam. The distance AB is measured by DE since the width of DE is due to the divergence of the direct beam. Whence $BD+AB/2+DE/2=BE$.

Having determined θ we find α by measuring $\alpha+\theta$. Tan$(\alpha+\theta)$ is equal to $BF+AB/2$ divided by the plate distance GA. In order to get $BF+AB/2$ it is necessary to add to BE which has been found already, the distance $EF+DE/2$.

Since the measurement of fine structure requires wave-length determinations for each component in the image F, the measurement of $EF+DE/2$ was carried out directly on the vacuum thermocouple densitometer. The width of the entrance slit in the diaphragm over the plate under investigation was 0.05 mm and this was therefore made the distance between successive plate settings in the line density measurements of the plates. To the moving part of the densitometer was attached a bar with fiducial mark which could be followed with the micrometer microscope and thus used to measure the distance that the plate moved.

In making a measurement the reflected line was placed in front of the densitometer slit. The plate was moved until the peak of the reflected line was found and the corresponding position of the fiducial mark was read on the micrometer. The plate was moved on its carriage until the diffracted line to be measured was near the slit. Readings on the galvanometer were taken every 0.05 mm from this point across the width of the diffracted line. Whenever the readings indicated a peak in the line, the position of the fiducial mark was read with the micrometer. The wave-length of each of these peaks was determined thus independently of the others in every case.

Curves produced by plotting the galvanometer deflections against wave-length proved to be reproducible in independent runs, thus suggesting the absence of any serious errors in this part of the work.

With the procedure described, measurements have been completed for the K lines of carbon, boron, oxygen and beryllium. These have been obtained in some cases in three orders. The combined effect of intensity of the line image and variation in dispersion for the different orders determined in

each case which order would give the best resolution of the fine structure under densitometer analysis.

Carbon

The lines of carbon were present on every plate although no carbon was placed on the anticathode. The source of this carbon is probably the grease vapor present at all times in the apparatus. In the 40 plates taken in this particular phase of the study (serial numbers 79 to 119) about 20 carbon lines were studied under the densitometer, the others being either too weak or too strong to give good resolution of the fine structure. Under visual observation most of the carbon lines appeared as continuous broad bands with maximum intensity on the short wave side. However, in several instances where small grazing angles were used, the first order carbon line appeared as a doublet, the shorter wave component being considerably the stronger.

A plot of the densitometer results obtained from a typical carbon line is shown in Fig. 2. Here the peaks indicate diminished transmission through the plates, i.e., the intensity maxima of x-radiation. It is apparent that here

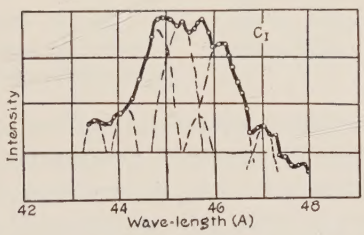

Fig. 2. Plot of densitometer results obtained from a typical carbon line.

we have a complex curve made up of several components. An attempt has been made to analyze this net curve into its constituents, a possible combination being shown in Fig. 2. An examination of this curve shows a rapid rise in intensity from the short-wave side to the first peak at 44.70A. A number of peaks follows with a gradual decrease in intensity to 46.20 where the curve again breaks in a sharp descent followed by a number of less prominent maxima. The small change of slope in the curve at 44.2A is a real effect and can be observed on all the curves of the carbon line. This demonstrates that the densitometer yields results sufficiently reproducible to attach a meaning even to a change of slope in the curve. The resolved lines shown in the figure are of course arbitrary to some degree. The position and magnitude of these components as shown do not represent the only way in which this curve could be built up. But little weight can be placed on the relative intensities of the minor components since that depends to a large degree on the method of choosing them.

The intensity of the line was found to depend on applied voltage. Although in general in soft x-ray spectroscopy the line intensity increases with

[13] Dershem, Phys. Rev. **34**, 1015 (1929).

the voltage and with increasing plate current, we find in the case of carbon that there is a decrease in line intensity with increase of these factors. This is in accord with the observations of Dershem[13] who explained the effect as due to the burning off of the carbon from the anticathode by the greater energy used. Thus only faint carbon lines appeared at 2700 volts.

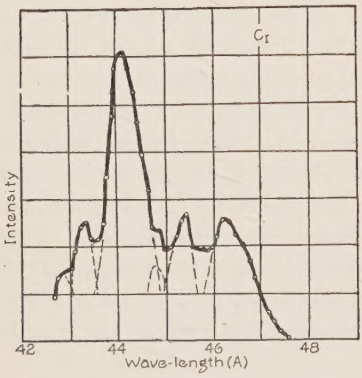

Fig. 3. Densitometer curve of the first order carbon line on a plate taken at 2300 volts.

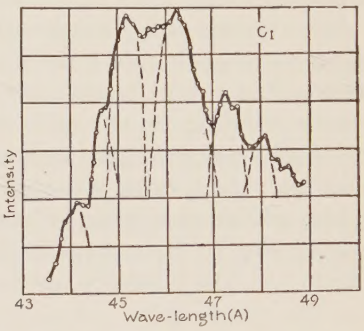

Fig. 4. Densitometer curve of the first order carbon line on a plate taken at 1500 volts.

It was found that the relative intensities of the components of the lines are dependent on the driving potential, the shorter wave components increasing in intensity relative to the long wave components at higher voltages. This interesting relation was specifically investigated by taking two series of plates under similar conditions excepting that the driving voltage for the one series was 1500 to 1600 volts and for the other series 2300 to 2800 volts.

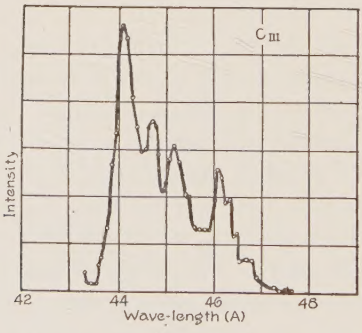

Fig. 5. Densitometer curve of a third order carbon line on a plate taken at 2500 volts.

Curves in Figs. 3 and 4 illustrate the different intensity distributions obtained in these tests. Fig. 3 is the densitometer curve of the first order carbon line on a plate taken at 2300 volts. This line under visual observation appeared as a doublet. A clearly defined peak is seen here at 43.3A. The curve then rises to a sharp maximum at 44.10A and shows two more distinct

peaks at 45.40A. Comparison with Fig. 2 shows that the small change of slope in that curve has now risen to the chief peak, the longer wave peaks being of less intensity. This is the case for all curves of lines taken at voltages of 2300 volts or more and therefore shows a shift of the maximum to the short wave side with increasing voltage.

Fig. 4 is a first order carbon line taken at 1500 volts. The principal peak is now at 45.3A with a second peak at 46.25. The short wave maxima have become reduced in intensity to mere kinks in the curve.

Fig. 5 is a densitometer curve of a third order carbon line showing unusually good resolution of the four principal maxima of the curve. This plate was taken at 2500 volts.

TABLE I

Plate No.	Order	Voltage	Wave-lengths of components of carbon.									
94	I	2300	42.70	43.23	44.10	44.70	45.4	46.25				
94	III	2300	42.90	43.24	44.00	44.65	45.25	46.00				
95	II	2300	42.60	43.06		44.48	45.28	46.20				
112	I	1900		43.45	44.05	44.62	45.35	46.20	46.55			
112	II						45.31		46.80			
97	I	1900				44.68	45.41	46.25		47.20	48.11	(weak)
113	II	1900			44.25	44.70	45.26	46.20	46.57	47.00		
114	I	1800			44.27	44.75	45.28	46.20		47.18	47.79	
114	II	1800			44.30	44.70	45.25					
116	I	1500			44.17	44.60	45.41	46.25		47.35	48.15	(weak)
115	I	1700				44.61	45.34	46.23		47.12	47.80	
115	II	1700	42.55	42.96	43.95	44.68	45.33	46.03		47.00		
93	I	2300	42.50	43.40	44.12	44.70	45.30					
96	I	1900			44.33							
79	III	2500		43.30	44.01	44.71	45.11	46.11	46.45	46.90	47.50	
87	I	1600	42.70	43.20	44.10	44.75	45.40	46.32	46.60	47.00		
Ave. values			42.66	43.23	44.05	44.66	45.33	46.20	46.60	47.21	47.70	

Taking all of the curves together there appears to be sufficient reason for concluding that the carbon K line contains 9 components centered about 42.7, 43.2, 44.0, 44.7, 45.3, 46.2, 46.6, 47.2, and 47.7A. We may also conclude that the relative prominence of these components and therefore the apparent position in the frequency scale of the unresolved line depends on the driving voltage, the maximum being 45.3A at 1500 volts and at near 44.0A at 2500 volts.

There is some uncertainty in the assignment of the title "$K\alpha$" to any particular component because of this shift in the maximum from 44.0 to 45.3A. Whatever the reason for the shift may be, it seems probable that the rate of decrease in intensity with decreasing voltage will be greater for the minor components than for the principal K line. Now since the 45.3A component was the maximum at 1500 volts, the lowest voltage at which lines could be obtained, it is perhaps justifiable to assign to the $K\alpha$ line of carbon the wave-length 45.33A.

No explanation can be offered at this time as to the significance of the fine structure here shown. Dr. C. B. Bazzoni has suggested that there is a possibility that the energy due to the excitation potentials of the other elements in combination with carbon interacts with the K-radiation of carbon to produce the satellite lines. The elements here in combination with the carbon are supposedly hydrogen and oxygen. It can be shown that the fre-

quency differences between the principal line and the various satellites are at least in qualitative agreement with this idea. There is also an agreement between the number of satellites observed and the number predicted from this consideration.

Boron

Boron lines were secured on 25 plates using boric acid, borax or boron on the anticathode. Fifteen of these lines were suitable for use in the densitometer. Second order boron overlapped the third order carbon line so that only the first order of boron could be measured.

The anticathodes of the boron compounds were prepared by fusing the compound on the anticathode. The line of pure boron was obtained by placing amorphous metallic boron obtained from Kahlbaum in a net-work of slots on the face of the anticathode.

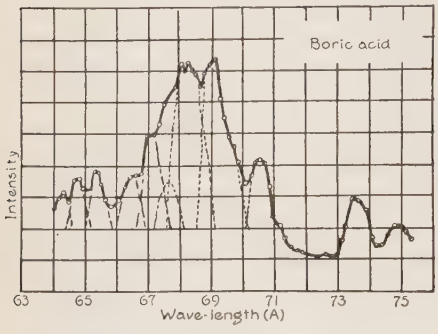

Fig. 6. Densitometer curve of a boron line taken with fused boric acid.

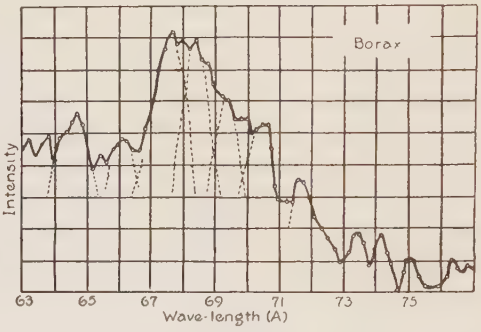

Fig. 7. Densitometer curve of a boron line taken with fused borax.

Under visual observation the $K\alpha$ line of boron appears as a continuous band broader than that of carbon. In several cases with small glancing angles the line appears to be just resolved into a doublet. The maximum of the line in general is on the short wave side. Two attendant lines on each side of the boron line are also visible to the eye in the spectra of boric acid and of borax. These attendant lines are absent in the spectrum of pure boron. This point was investigated carefully and plates from pure boron were obtained with lines even denser than the lines of the compounds with however no trace of satellites.

For given conditions of exposure the more intense lines of boron were obtained at higher voltages. Most of the lines of boron were obtained at 2300 volts more.

Fig. 6 shows a densitometer curve of a boron line taken with fused boric acid. This curve was resolved into components in a manner similar to that described for carbon. Fig. 7 is the plot of a line taken with fused borax on the anticathode. The conditions under which these plates were taken and the values of the resolved components are given in Table II

TABLE II.

Plate No.	Material	Voltage	Plate Distance	Wave-lengths of components of boron												
81	Borax	2500	30cm	64.85		66.43	67.37	68.28	69.30	70.30	71.63		73.26	73.90	75.00	76.60
83	"	2300	30	64.50			67.30	68.12	69.15	70.22	71.20	72.67		73.75	74.90	
102	"	1870	30	64.81		66.40	67.30	68.30	69.33	70.00	71.26	72.24	73.10		74.90	77.00
102(2)	"	1870	30			66.89	67.40	68.30	69.35	70.00	71.25	72.00				77.10
88	"	1850	30	64.72		66.70	67.70	68.40	69.38	70.20	71.20					76.46
68	Boric acid	2500		64.85	65.25	66.57	67.87		69.33	70.40	71.24	72.05	73.28	74.08	75.0	77.00
71	"	2500		64.75		66.40	67.52	68.10	69.30	70.40	71.25	72.00	73.28	74.13	75.0	
72	"	2500		64.40	65.69	66.49	67.40	68.11	69.07	70.51	71.20					
96	"	1900	30	64.40		66.52		68.14	69.10	70.20	71.50	72.17	73.36			76.45
97	"	1900	30		65.30	66.00	67.70		69.40			71.90	73.19	74.38		
73	Boron	2500	40				67.87		69.00							
74	"	2500	30				67.80	68.49	69.16	70.74						
94	"	2300	30				67.40	68.16	69.41	70.42						
95	"	2300	30				67.70	68.34	69.42	70.41						
114	"	1800	40				67.76	68.38	69.45	70.30						
Averages				64.68	65.45	66.55	67.61	68.38	69.33	70.36	71.32	72.03	73.20	74.00	74.95	76.75

No marked difference is observed between the fine structure of the borax lines and the boric acid lines. The densitometer curves of pure boron are shown in Figs. 8 and 9. It is seen that but four components remain in the lines in this case. It would appear, therefore, that the presence of the attendant lines may be due to the association of the boron with other elements.

The curve of Fig. 8 is for a line taken at 2300 volts while Fig. 9 shows a curve of a line taken at 1800 volts. As in the case of carbon, a definite shift of the maximum to the short wave side occurs with increasing voltage. Reasoning in a manner similar to that used in selecting the wave-length of the $K\alpha$ line of carbon it would appear justifiable to assign a wave-length 69.33A to the $K\alpha$ line of boron.

The results in Table II seem to show that there are 13 components in the lines obtained from the boron compounds located at 64.68, 65.45, 66.55, 67.61, 68.38, 69.33, 70.36, 71.32, 72.03, 73.20, 74.00, 74.95, and 76.75A and that the spectrum of the K-radiation of the boron used contains four components at 67.61, 68.38, 69.33 and 70.36A. All of these figures are uncertain in the second decimal place.

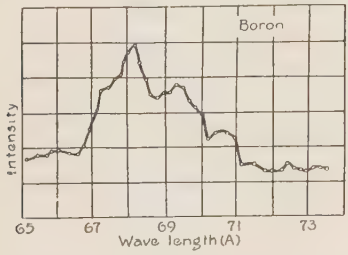

Fig. 8. Densitometer curve of pure boron line taken at 2300 volts.

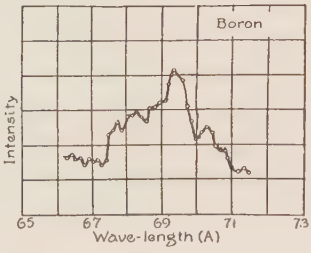

Fig. 9. Densitometer curve of pure boron line taken at 1800 volts.

It should be noticed that although the amorphous boron was "metallic" undoubtedly it contained appreciable percentages of oxide, hydride and probably nitride.

The explanation of the fine structure through the hypothesis of the development of addition and subtraction quanta through the interaction of the boron radiation with excited or neutral atoms associated in the molecular lattice is more difficult than for carbon. If we accept these results as showing that the K-radiation of boron is composed of four components, then to predict the fine structure of the compounds of boron on the above hypothesis, it is necessary to calculate these addition and subtraction quanta for each of the four compounds. Boron is associatd with oxygen in fused boric acid and with sodium and oxygen in fused borax.

These calculations being carried out the number of satellites predicted is found to be substantially the same as the number observed. Also in many cases a close concordance between predicted and observed wave-length is found. However, due to the fact that the lines predicted due to sodium are in all cases coincident within 0.1A with oxygen lines or original component lines no definite conclusions can be formulated at this time.

There can be no doubt that these attendant lines are associated with the $K\alpha$ of boron. They do not seem to be due to any other element. In no case was it found possible to secure the attendant lines in the absence of the boron line. It is of some significance that in every case where the attendant lines appeared the oxygen line was present also.

Oxygen

The $K\alpha$ line of oxygen appears on all plates of the boron compounds and on many others. Although several densitometer curves were taken of this line, only a single component appeared. Fig. 10 shows a curve of a typical oxygen line. The wave-length assigned to $K\alpha$ oxygen is 23.77 ± 0.06A.

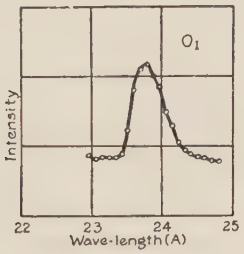

Fig. 10. Densitometer curve of a typical oxygen line.

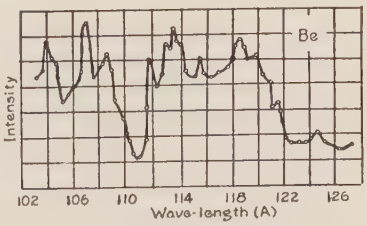

Fig. 11. Densitometer curve of the lines of beryllium.

Beryllium

A target of pure beryllium obtained from the Beryllium Company of America was used in the measurements of this element. Only faint traces of the presence of a line with three components could be obtained at 1800 volts using currents as large as 150 milliamperes for six hours. At 2700 volts beryllium lines of more intensity could be obtained using 40 milliamperes for two and a half hours. The line appears as a broad band with two distinct maxima. On the short wave side of this band a distinct line appears of the same intensity as the band.

Fig. 11 shows a densitometric curve of the lines of beryllium. Only four plates have been taken of this element of which three are suitable for use in the densitometer. It is not believed to be justifiable, therefore, to deduce values for the fine structure of the lines, but only for the three principal peaks. These maxima occur at 107.2 ± 0.4A, 113.2 ± 0.3A and 118.7 ± 0.2A. The great breadth, approximately 15A, of this line is peculiar to this element and should be remarked.

Discussion

It will be observed that the value we obtain for oxygen (23.77 ± 0.06A) is in good agreement with the value found by Söderman (23.77 ± 0.08A). Our value for beryllium (113.2 ± 0.3A) also checks with Söderman's value (113.4 ± 0.3) measured to the easily distinguishable maximum of this line.

The method of measurement used in our work in which a single component is selected from a group would seem to explain the divergence of our values for carbon and boron from those obtained by other investigators. Thus a visual measurement to the middle of the carbon line yields a value of 44.65A, in good agreement with the value of Söderman (44.70) and of Howe (44.62). The value (45.33) however, which we have finally selected based on a study of the components agrees very closely with the determination of Weatherby (45.4A). Similarly in the case of boron, a visual measurement to the middle of the line gives 67.8, in good agreement with Söderman's value (67.80 ± 0.2A) and with that of Thibaud (68.0A) while the value here selected for the principal maximum is considerably higher, namely 69.33A.

The great breadth of the carbon, boron and beryllium lines must be accepted as a real effect, for the $K\alpha$ line of oxygen at 23.77A and the N doublets of tungsten at about 56A and 60A are fine lines.

In conclusion the author wishes to thank Dr. B. B. Weatherby who aided him in beginning this work. And especially does the author wish to express his thanks to Dr. C. B. Bazzoni under whom this work was carried out and whose many helpful suggestions and keen interest are largely responsible for the success of these measurements.